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Improving the properties of ceramic foams by a vacuum infiltration process

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Abstract

Reticulated ceramic foams are widely used for industrial applications such as metal filtration, exhaust gas and air purification, catalyst support and others. In this work, the compression strength and specific surface area of reticulated foams have been improved, while at the same time maintaining a high level of permeability in the final foam structure. In particular, a vacuum infiltration step by using a suitable slurry, followed by a pre-sintering cycle was adopted for filling up the hollow struts, generated due to the burnout of the PU foam. Furthermore, various mixtures of fine and coarse-grained alumina as well as in combination with zirconia, were utilised with the aim of controlling the foam properties such as compression strength, specific surface area and permeability. The compression strength was improved by a factor of two for alumina foams by infiltrating the hollow struts, and by a factor of four when infiltrating the struts of ZTA foams, with the composition 70 mol% Al_2O_3 and 30 mol% ZrO_2. The weight gain resulting from the vacuum infiltration process was in the order of 10 wt%.

Keywords: Al_2O_3; ZTA; Reticulated foams; Vacuum infiltration; Strength

1. Introduction

Reticulated ceramic foams of diverse compositions (e.g. alumina, SiC, cordierite, etc.) produced by the polymer sponge (replica) method possess beneficial properties such as an open, three-dimensional network structure with an interconnecting porosity in the range of 75 to >90 vol%, a high inner geometric surface area, low density, good permeability and a low pressure drop.[1–3] Consequently, these porous foam structures have low resistance to fluids or gases, and a turbulent flow regime is generated due to the tortuous flow path within the material.

Ceramic foams with a homogeneous cell distribution and modified surface structures are very promising carriers for catalyst supports, as they easily can be coated with high surface area wash coats and catalytic components by well established methods.[4] In relation to extruded honeycomb catalyst supports, the tortuous flow path through a ceramic foam substrate is expected to enhance the gas–solid heat- and mass-transfer, leading to high reaction rates per unit volume.[5] The fluid dynamic behaviour of ceramic foams in automotive exhaust catalysts has been assessed in a previous work.[6]

Due to the presence of hollow struts in foam ceramics induced by the replica method, relatively poor mechanical properties are typically achieved,[2] but strength can be increased by modifying processing parameters such as slurry viscosity. This however leads to an increase in thickness of the struts and thus a higher relative density of the ceramic foams, resulting in a decrease in permeability (higher pressure drop), as the build-up of material

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at the struts forms dense walls which reduce the interconnections among the cells.

The aim of this work was the fabrication of alumina based ceramic foams with an open pore structure, improved mechanical strength and micro-porous struts, which increase the specific surface area of the foam structures and thus enhance their catalytic activity. Alternatively, zirconia-toughened alumina (ZTA) based foams were manufactured, as ZTA is known to possess a high strength due to the transformation toughening mechanism.[7]

By this approach, highly porous ceramic foams with suitable mechanical strength and improved surface characteristics for enhancing gas–solid reactions were achieved. For adjusting the active surface area, several compositions with different ratios of fine- and coarse-grained alumina powders were investigated. The foams were tested with respect to parameters such as mechanical strength, bulk- and skeleton density and specific surface area, taking into account the requirements for catalytic applications, in particular the generation of turbulent gas flows.

In order to achieve a low pressure drop,[8] a filigree strut architecture is desirable in combination with a mechanical strength sufficient for engineering application. In particular for the applications as catalyst support for automotives, porous ceramics have to withstanding stress during assembling and long term operation, as they have to guarantee an adequate operation lifetime.

For increasing the foam strength and coincidentally improving both, the macro- and microstructure, a vacuum infiltration process was applied to infiltrate the hollow struts which are formed after burnout of the PU scaffold as consequence of the polymer sponge (Schwartzwalder) method.[9]

2. Experimental procedure

2.1. Ceramic foam preparation and characterization

Alumina and ZTA foams were fabricated by the replica method, as described in detail elsewhere.[5,9] For foam preparation, a polymeric sponge of polyurethane (PU) was impregnated with a ceramic slurry and dried at room temperature for 24 h. The polymer sponge was subsequently burned out and the remaining ceramic struts sintered to a porous ceramic scaffold with an open porous structure. For modifying the specific surface area (BET) and the mechanical properties (compression strength), different mixtures of coarse and fine grained alumina was investigated and the addition of zirconia for a ZTA composition examined. For preparing the ceramic slurry, alumina (CT 3000 SG, Almatis, Germany), with a particle size D 50 of 0.8 μm and a BET of 7.5 m^2/g was used as fine alumina. For adjusting the above mentioned properties, coarse-grained alumina (T 60, Almatis, Germany) with a particle size D 50 of 0.2–0.6 mm and a BET <1 m^2/g was investigated. The amount of CT 3000 SG and T 60 were varied from 0 to 100 wt%. For the preparation of zirconia-toughened alumina foams (ZTA), a ceramic slurry with an Al_2O_3/ZrO_2 molar ratio of 70:30 were prepared (AZ73) by co-milling of the submicron alumina CT 3000 and yttria-stabilized zirconia powder with a particle size D50 of 230 nm and a BET of 14.5 m^2/g (TZ-3Y, Tosoh, Japan). For preparing the ceramic slurry, the alumina respectively zirconia powder was added to demineralised water by adjusting a solid load of 80 wt%. As deflocculating additive, 0.84 wt% Dolapix CE 64 (Zschimmer & Schwarz, Germany) was used in order to ensure an adequate rheological behaviour. Deagglomeration and mixing of the ceramic slurry was carried out by conventional ball milling technique on a roller mill (ZOZ, RM1, Germany) in polypropylene bottles for 24 h, using YTZ ceramic balls of 10 mm diameter (Tosoh, Japan). As binder, 1.5 wt% Polyvinylalcohol PVA (Optapix PA 4G, Zschimmer & Schwarz, Germany) was added after the milling procedure. For this purpose, the ceramic slurry was subsequently heated up to 90 °C for 2 h by constantly stirring. Several drops of Contraspum KWE (Zschimmer & Schwarz, Germany) were added as anti-foaming agent during the cooling process, and the slurry mixed again for ca. 1 h until room temperature was reached. The as prepared ceramic slurry was adjusted to a viscosity of $\eta = 0.405\ \mathrm{N\,s\,m^{-2}}$ (Viscosimeter PHYSICA RHEOLAB MC 120).

Organic polyurethane (PU) sponges of 8, 10 and 15 ppi (pores per inch) (FoamPartner-Fritz Nauer AG, Switzerland) were used as skeletal structures, as they possess an adequate resiliency and a hydrophobic behaviour, as well as a complete volatilisation at 600 °C. By assuming an isotropic cell structure, the pore per inch values correspond to approximately 64 (8 ppi), 100 (10 ppi), and 225 (15 ppi) cells per square inch (cpsi), and thus to a mean pore volume, V_{pore}, of 0.032, 0.0164 and 0.0049 cm^3, respectively.[6]

The PU foams were dipped into the as prepared ceramic slurry, compressed to fill up the void space and squeezed between two PU foams to remove excess ceramic slurry. Remaining closed pores were eliminated by blowing carefully compressed air through the foam structures. The slurry coated PU foams were dried at room temperature for 24 h on alumina coated SiC firing auxiliaries.

Burning out of the PU foam and sintering of the cellular structures was realised in one single step under atmospheric conditions in the same furnace (CeramAix, D-5100/Aachen, Germany). To avoid thermo induced cracks of the sensitive porous structures, a heating rate of 60 °C/h was chosen for decomposing the organic PU foam between 260 and 600 °C and further to the final sintering temperature of 1200, 1300 or 1400 °C, respectively. The holding time at sintering temperature was 2 h, the cooling rate 120 °C/h to 800 °C and 300 °C/h to room temperature in order to avoid thermal induced cracks.

The principle shrinkage behaviour of the various compositions was determined by dilatometer experiments on samples of 5 mm × 5 mm × 10 mm (Bähr, DIL802, Germany). The bulk density, and consequently the total porosity of the foam structures, was calculated by the weight to volume ratio. For determining the skeleton density, the foams were crushed and the density of the solid struts measured by helium pycnometry (Micromeritics Accu Pyc 1330, Germany).

Specific surface area (SSA) measurements were carried out by the gas adsorption method (BET, Coulter® SA3100™), which assures a measuring range from 0.1 to 2000 m^2/g. The strut morphology was investigated by scanning electron microscopy (SEM, TESCAN Vega TS 5136 MM, Czech Republic).

Mechanical tests on the sintered foams were performed by determining the compression strength with a Zwick Universal-Proof machine Z005 fitted with a 5 kN measuring cell, using a crosshead speed of 1 mm/min. For this purpose, samples of the size 50 mm × 50 mm × 18 mm were positioned between two aluminium plates (60 mm × 60 mm × 15 mm). Due to the irregular surface structure of the foams, a rubber mat of 5 mm thickness was placed between the aluminium plates and the ceramic foams to ensure a uniform loading on the foam surface and to eliminate surface edge fracture initiation. For each test compressive load vs. crosshead displacement was recorded, and eight specimens per type were measured for calculating the standard deviation.

2.2. Vacuum infiltration of pre-sintered foams

As ceramic foams produced by the polymer sponge method are quite fragile and possess only a moderate mechanical stability, a vacuum infiltration process was established to infiltrate the hollow struts after pre-sintering the ceramic foams at 1200 or 1400 °C, respectively. With this procedure, the hollow struts of the ceramic skeleton could be filled up and after a second sintering process the compression strength considerably increased. For this purpose, a ceramic slurry of the same principle recipe as for the ceramic foams was prepared, but with only CS 3000 SG, without binder and with a solid load of only 65 wt%, leading to a lower viscosity and thus enable a proper infiltration of the hollow struts. For the infiltration procedure, the sintered foams were placed in a vacuum tight glass cylinder and a vacuum of 0.5 mbar applied (vacuum pump SPEED/VAC Model ED 100). The ceramic slurry was poured through a funnel, positioned above the glass cylinder, and introduced into the glass cylinder until the foam was totally covered. After a dwell time of 5 min, the vacuum pump was switched off in order to avoid water losses due to evaporation, leading to an increasing of the pressure to 11 mbar. To avoid an additional coating of the struts, causing an undesirable increase in strut thickness, the infiltrated foams were flushed with water and subsequently dried at room temperature for 24 h. The as infiltrated foams where subject to a second sintering procedure at 1200 or 1400 °C, respectively and the weight increase after infiltration was determined.

3. Results and discussion

3.1. Effect of alumina powder grain size vs. foam properties

The effect of several fractions from CT 3000 SG (fine) and T60 (coarse) alumina powders, as well as the influence of the sintering temperatures between 1200 and 1400 °C were investigated concerning shrinkage behaviour, microstructure formation, specific surface area and compression strength. The shrinkage behaviour of samples with 20 wt% 3000 SG – 80 wt% T60 (sample C) and 100 wt% CT 3000 SG (sample D) was investigated by dilatometry from RT to 1500 °C. While sample D (100 wt% CT 3000 SG) shows a shrinkage rate of 18%, the shrinkage rate of sample C (80 wt% T60) was only 3% (Fig. 1). This is obvious due to the fact that the fine alumina powder (CT 3000 SG) has a much higher sintering activity in relation to the coarse powder T60. Due to the very low sintering activity of the coarse alumina particles (T60), they are subject to an insignificant shrinkage and thus acted as “space holder” which are blocking the shrinkage. Hence, CT 3000 SG is the “binding agent” between the coarse alumina particles (T60). SEM micrographs of the microstructure of the samples A (100 wt% T60), B (10 wt% CT 3000 SG, 90 wt% T60), C (20 wt% CT 3000 SG, 80 wt% T60) and D (100 wt% CT 3000 SG), sintered at 1400 °C, are reported in Fig. 2. The micrographs confirm the effect of different particle sizes with respect to the sintering behaviour, porosity, specific surface area and compression strength. While an increasing fraction of fine alumina leads to a decreasing assemblage of macro-pores in the 1–3 μm range, an increase in total density of the struts and consequently the fraction of micro-pores with submicron size was observed.

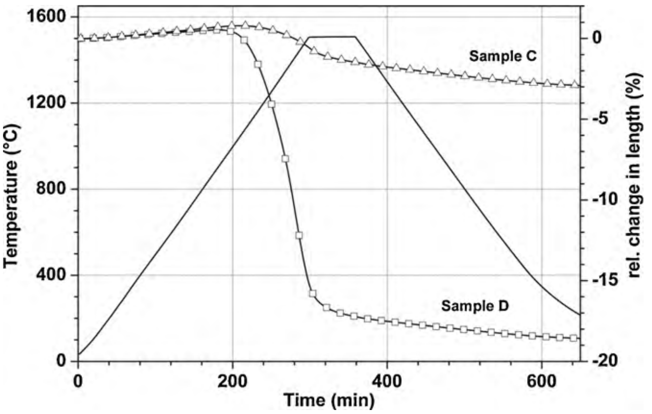

Fig. 1. Shrinkage behaviour of fine grained alumina CT 3000 SG (sample D) and a mixture of 20 wt% CT 3000 SG and 80 wt% T 60 (sample C), heated up to 1500 °C.

The influence of the fine- to coarse-grained ratio of alumina regarding the specific surface area (BET) values, as a function of the sintering temperature, is shown in Fig. 3. For a sintering temperature of 1200 °C, a linear increase in BET with increasing fine alumina fraction (CT 3000) is obvious, due to the high specific surface area of CT 3000 SG (Fig. 1). For sintering temperatures of 1300 and 1400 °C, the slope of the BET is non-linear with increasing fine alumina fraction due to grain growth effects and a higher shrinkage rate. This leads to higher skeletal densities of the foam struts coevally with an increasing strength (Fig. 4).

Investigations concerning the compression strength before infiltration of the hollow struts show an increase in strength with increasing fraction of CT 3000 SG and with increasing sintering temperature. For foams of 100 wt% CT 3000 SG (sample D), the strength values were 0.21 MPa at 1200 °C, 0.26 MPa at 1300 °C and 0.42 MPa at 1400 °C sintering temperature (Fig. 4), for ceramic foams of only T 60 (sample A), the strength was negligible, independent on the sintering temperature. For the different mixtures of fine and coarse alumina it can be stated, that lower sintering temperatures represent higher specific surface areas, but considerably lower compression strength. The increase in strength with rising fraction of fine alumina is in agreement with the increase of strut (skeleton) density and strength.

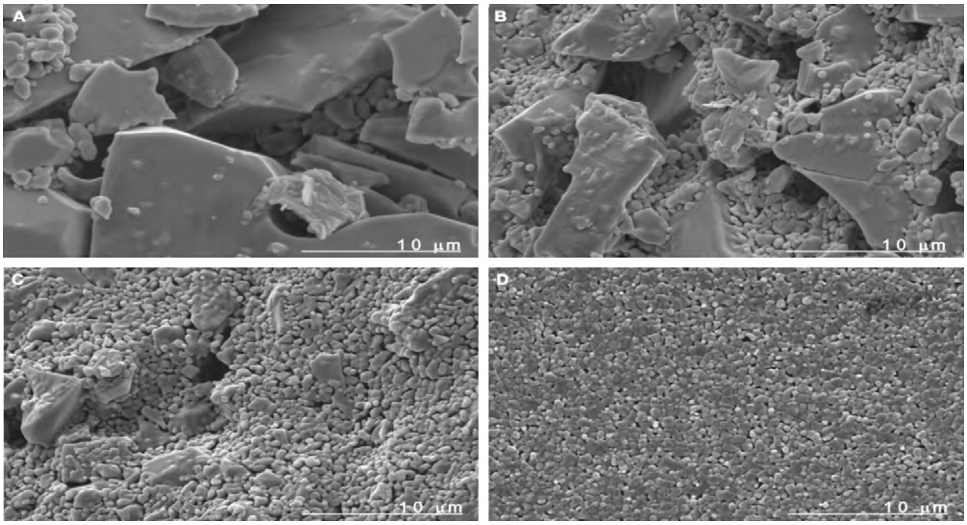

Fig. 2. Microstructure of alumina sintered at 1400 °C with different fractions of fine (CT 3000 SG) and coarse (T60) alumina: (A) 100 wt% coarse; (B) 10 wt% fine – 90 wt% coarse; (C) 20 wt% fine – 80 wt% coarse; (D) 100 wt% fine alumina.

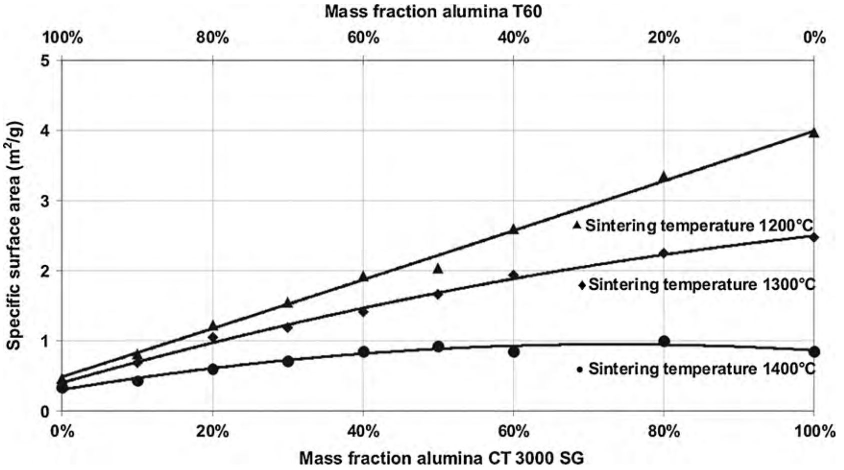

Fig. 3. Specific surface area (BET) vs. mass fraction of fine (CT 3000 SG) and coarse (T60) grained alumina, as a function of the sintering temperature.

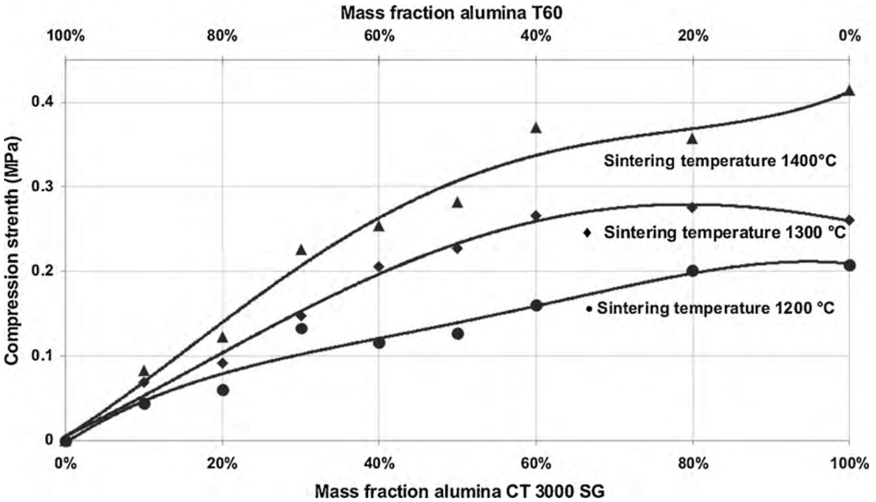

Fig. 4. Compression strength vs. sintering temperature for foams of the bimodal system CT 3000 SG and T60 before infiltration of the struts.

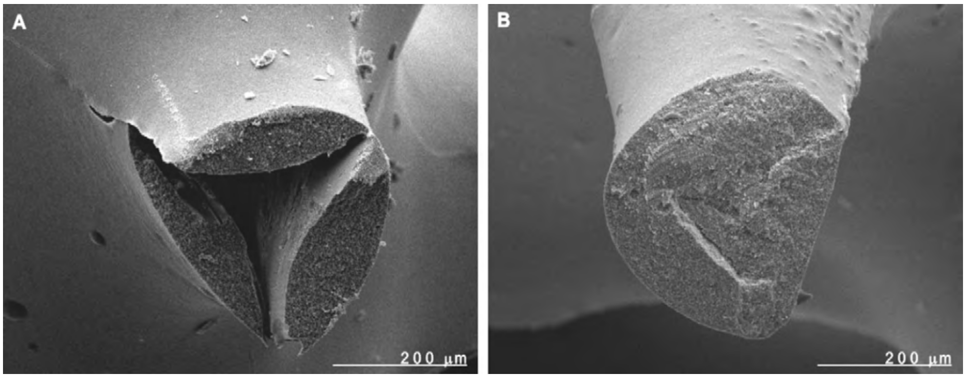

Fig. 5. (A) Hollow strut after burnout of the PU foam and (B) dense strut after vacuum infiltration, sintering temperature 1400 °C.

The determined strength values in this work are somewhat lower than those typically reported for reticulated alumina foams, which range from 0.4 to 2.5 MPa.[10–12] But it has to be noted that most of those data were collected by testing commercially available foams, which usually exhibit denser struts due to sintering aids for reducing the sintering temperature. Reaction-bonding also is an alternative approach to increase the strength of reticulated foams.[13] The foams produced in our work possess in generally higher total porosity (>90 vol%) and no additional sintering aids were used in order to preserve the micro-porosity of the struts for retaining the specific surface area. Higher sintering temperature yield to increasing strength, due to denser ceramic struts which is in accordance with literature.[14]

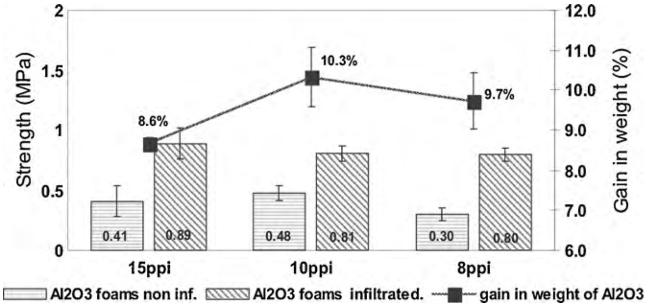

Fig. 6. Compressive strength with standard deviation and weight gain before and after vacuum infiltration of pure alumina foams (sample D), sintered at 1400 °C.

3.2. Vacuum infiltration of pre-sintered foams

A factor limiting the strength of reticulated porous ceramics is the presence of triangularly shaped holes inside the struts, leading to stress concentration at the edges, as well as cracks and other defects deriving from deficient coating of the PU sponge by the ceramic slurry.[2] Indeed, failure mainly originates near the apex of the triangular holes in the struts, where the thickness of the ceramic coating on the PU template is limited. Due to this effect, several authors have suggested additional coatings of pre-sintered foams, in order to cover the defects and increase the strut thickness.[15,16] However, with this approach the triangular channels remain within the struts and the struts have a considerably increased thickness which will significantly reduce the permeability of the reticulated structures. Jun et al.[17] proposed an infiltration of the struts by a ceramic slurry and pre-sintering at 800 °C. However, in that work only a very limited densification effect of the struts was achieved and the increase in strength was mainly due to the rounding effect of the sharp edges on the strut inner holes. This was due to covering pre-existing flaws, but without a complete infiltration of the hollow struts. In the same work, it was demonstrated that an additional vacuum infiltration can lead to a significant improvement in strength in relation to a simple second coating process of pre-sintered foams.

Fig. 5 shows SEM images on the foam structure before and after infiltration of the hollow struts, the micrographs clearly shows that a complete infiltration of the struts could be achieved by the vacuum infiltration process. The application of vacuum led to a decidedly better degree of strut densification with respect to the results reported by Jun et al.[17] In order to limit the increase in foam density after infiltration due to rising strut thickness, the slurry layer deposited on the strut surface was removed by gently rinsing the foams in water after vacuum infiltration. The measured weight gain after infiltration could be determined to be in the order of 8–10 wt% (Figs. 6 and 7).

The skeleton density of alumina foams of 8 ppi was determined by helium pycnometry to be 3.92 g/cm^3. The bulk density (weight/volume ratio) for the alumina foams before infiltration was determined to be 0.297 g/cm^3, corresponding to a total porosity of 92.4%. After slurry infiltration the bulk density increased to 0.325 g/cm^3, related to a total porosity of 91.7%.

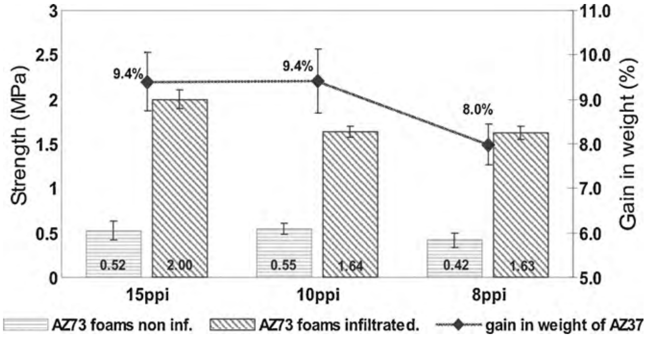

Fig. 7. Compressive strength, with standard deviation, and weight gain before and after vacuum infiltration, for ZTA foams (Al_2O_3–ZrO_2 = 70:30 mol%), sintered at 1400 °C.

By adjusting the slurry characteristics for vacuum infiltration (solid loading and viscosity) as well as the subsequent sintering temperature, we were able to achieve a considerable improvement in strength by the vacuum infiltration process. The experiments for determining the mechanical properties of the foams were carried out on samples sintered at 1400 °C for 2 h, before as well as after vacuum infiltration.

Fig. 6 reports the compressive strength for foams of 100% fine alumina (sample D) of 8, 10 and 15 ppi, before and after vacuum infiltration as well as the weight gain. The average strength after vacuum infiltration was 0.9 MPa (15 ppi), 0.81 MPa (10 ppi) and 0.80 MPa (8 ppi), the increase in strength was more than 100% for the 15 ppi and 8 ppi foams and just below 100% for the 8 ppi foams. No big variation in strength or weight gain was observed as a function of the cell size, the measured weight gain was 8.6% (15 ppi), 10.3% (10 ppi) and 9.7% (8 ppi).

3.3. Zirconia toughed alumina foams (ZTA)

Supplementary investigations where carried out to improve the mechanical properties of reticulated foams by the addition of zirconia (30 mol% 3Y-TZP, 70 mol% CT 3000 SG), leading to ZTA (zirconia-toughened alumina) foams.

The weight gain due to vacuum infiltration was in the same order as for alumina foams: 9.4% (15 ppi), 9.4% (10 ppi) and 8.0% (8 ppi).

In Fig. 7 the strength values before and after infiltration of the ZTA foams with ZTA slurry (sample AZ73) and sintered at 1400 °C, as well as the weight gain is reported.

While the strength before infiltration was of the same order than that of the pure alumina foams (Fig. 6), the strength after infiltration with ZTA slurry is considerably increased: (+384%, 15 ppi), 1.65 MPa (+315%, 10 ppi) and 1.62 MPa (+388%, 8 ppi), respectively. This can be expressed mainly by the stress-induced phase transformation from tetragonal zirconia into the more stable monoclinic form.[7,18,19]

The skeleton density of the ZTA foams of 8 ppi was determined by helium pycnometry to be 4.47 g/cm^3. The bulk density (weight/volume ratio) for the ZTA foams before infiltration was determined to be 0.395 g/cm^3, corresponding to a total porosity of 91.1%. After slurry infiltration the bulk density increased to 0.436 g/cm^3, related to a total porosity of 90.2%.

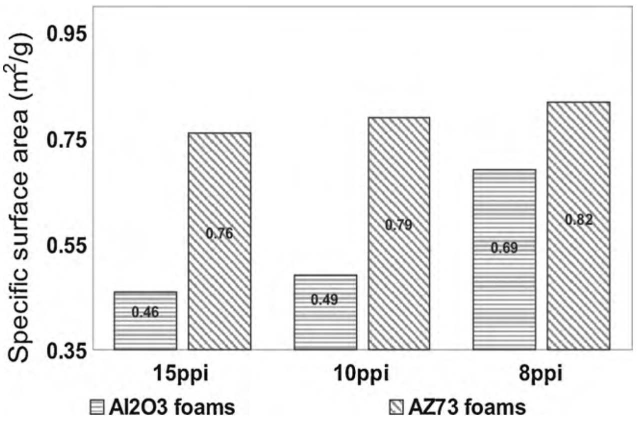

Fig. 8. Specific surface area (BET) for Al_2O_3 and Al_2O_3–ZrO_2 (70:30) (AZ73) foams sintered at 1400 °C after vacuum infiltration.

The specific surface area for the AZ73 samples remained higher for all cell sizes, with respect to foams consisting of 100% fine alumina (Fig. 8), what can be justified due to the higher specific surface area of the corresponding zirconia powder TZ-3Y (14.5 m^2/g), in relation to the alumina powder CT 3000 SG (7.5 m^2/g).

4. Conclusions

It could be shown that an increase in the mass fraction of fine alumina (CT 3000 SG) vs. coarse alumina (T 60) leads to an increase in shrinkage, specific surface area and strength for replica ceramic foams. An increase in sintering temperature is reducing the specific surface area (BET). At low sintering temperature, the high specific surface area of the fine alumina powder prevailed, while at higher temperature the sintering- and grain growth effects are dominating.

Due to the new developed vacuum infiltration process, the hollow struts of the replica foams, which originate from the burnout of the PU template, could be filed up completely. This results in a considerable increase in compression strength in the range of 100%, associated by a weight gain of ca. 10%.

ZTA foams showed similar strength values as alumina foams before vacuum infiltration. Due to the vacuum infiltration procedure, the strength increases considerably up to 388%, combined with a weight gain of ca. 9 wt%.

In this work, we showed the clear benefits of applying a vacuum infiltration process to improve the properties of replica ceramic foams by refilling the hollow struts. Furthermore the addition of zirconia, resulting in ZTA ceramics, leads to a considerable improvement in strength by maintaining a higher specific surface area.

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